FCS21 - Procedure for Uncertainty in Measurement

Table of Contents

1. Scope

1.1. This procedure outlines the practices for the FCU to determine Uncertainties in Measurement for values that contain a quantifiable uncertainty for reported measurements.

2. Background

2.1. To establish the practices for documenting the examination of evidence to conform to the requirements of the Department of Forensic Sciences (DFS) Forensic Science Laboratory (FSL) *Quality Assurance Manual*, the accreditation standards under ISO/IEC 17025:2017, and any supplemental standards.

2.1.1. The FCU is not a calibration laboratory or a testing laboratory that performs its own calibrations. The laboratory uses outside vendors to perform calibrations. These vendors shall have and apply a procedure to estimate the uncertainty of measurement for all calibrations and types of calibrations. Conformance shall be accomplished as per FCU-specific SOP's.

2.1.2. Standard operating procedures (SOP), when applicable, will include considerations for estimating the uncertainty of measurement. If the nature of the examination procedure precludes a metrologically and statistically valid calculation of uncertainty of measurement, the units will

attempt to identify all the components of uncertainty and produce a reasonable estimate.

3. Safety

3.1. Not applicable

4. Materials Required

4.1. Not applicable

5. Standards and Controls

5.1. Not applicable

6. Calibration

6.1. Not applicable

7. Procedures

7.1. The FCU will identify each SOP in which uncertainty of measurement may be reported. If a quantitative numerical measurement result is included in an FCU Report of Examination, the uncertainty of measurement must be reported. Additionally, an FCU Report of Examination must not give a wrong impression of the uncertainty of measurement.

7.1.1. Example processes that require a determination of the uncertainty in measurement include weight determination using balances and heroin purity using gas chromatography flame ionization detection (GC-FID). Refer to specific SOPs for further clarification.

7.1.2. Example calculations for an uncertainty measurement are provided in the Appendix.

7.1.3. Uncertainties must be calculated on an annual basis, or more, as directed by the Technical Lead, or designee.

7.2. All units reporting quantitative test results will have and apply a procedure to estimate the uncertainty of measurement. The procedure for estimation of measurement uncertainty:

7.2.1. Requires the specific measuring device or instrument used for a reported test result to have been included in or evaluated against the estimation of measurement uncertainty for that test method.

7.2.2. Includes the process of rounding the expanded uncertainty.

7.2.3. Requires the coverage probability of the expanded uncertainty to be a minimum of **95.45%.**

7.2.4. Specifies the schedule to review and/or recalculate the measurement uncertainty.

7.3. Records will be maintained by the FCU to describe the process used to develop the estimation of uncertainty. Estimates of uncertainty of measurement must be available for review when any of the following conditions exist:

7.3.1. The measurement of uncertainty is relevant to the validity or interpretation of the examination results.

7.3.2. The measurement of uncertainty is required by the contributor.

7.3.3. The measurement of uncertainty affects compliance to a specific limit.

7.4. Estimation of measurement of uncertainty will be based on knowledge of the performance of the method, previous experience and validation data as well as any significant parameters that affect the measurement result as per unit-specific SOP's.

7.4.1. The following records are maintained for each estimation of measurement uncertainty:

7.4.1.1. A statement defining the measurand,

7.4.1.2. A statement of how traceability is established for the measurement,

7.4.1.3. The equipment (*e.g.* measuring device(s) or instrument(s) used),

7.4.1.4. All uncertainty components considered,

7.4.1.5. All uncertainty components of significance and how they were evaluated,

7.4.1.6. Data used to estimate repeatability, intermediate precision, and/or reproducibility,

7.4.1.7. All calculations performed,

7.4.1.8. And combined standard uncertainty, coverage factor, coverage probability, and resulting expanded uncertainty.

7.5. Additional Report of Examination Guidelines

7.5.1. An FCU Report of Examination may include additional information, deviations from, additions to, or exclusions from the test method and information on specific test conditions such as statement on the uncertainty of measurement or a statement of the compliance / non-compliance to the standards when it is necessary for the interpretation of the examination results according to the *FCS02 – SOP for General Laboratory Procedures for FCU*.

7.5.2. The estimated uncertainty is required to be included in the test report when it impacts the evaluation of a specification limit stated by a regulatory body, a statute, case law, or other legal requirement. When reported, the uncertainty statement is required to include the measured quantity value, **y**, along with the associated expanded uncertainty, **U**, and the coverage probability and in the format of **y ± U** and the units of y and U to be consistent. The rounded expanded uncertainty is limited to at most two significant digits, unless there is documented rationale for reporting additional significant digits; and the rounded expanded uncertainty is to be reported to the same level of significance as the measurement result.

8. Sampling

8.1. Sampling plans are required when taking part of a representative sample of a substance, material or item to provide for testing and reporting on the whole substance, material or item.

8.2. Each casework unit, when necessary, will have plans and procedures for the sampling of evidence included in the appropriate SOPs (see *FCS02 – SOP for General Laboratory Procedures for FCU*). Sampling plans will be based on statistical methods when reasonable. The sampling plans will address the factors to be controlled to ensure the validity of the examination results.

8.2.1. At a minimum, unit sampling plans will ensure:

8.2.1.1. An evaluation of the selected population for homogeneity is performed.

8.2.1.2. The population has a reasonable expectation of homogeneity.

8.2.1.3. The sampling plan makes use of probability and provides and opinion or interpretation with a minimum confidence level of 95% (specifically, **95.45%**).

8.2.1.4. Each item selected meets the sampling plan level of confidence to be tested completely.

8.2.1.5. Appropriate action is taken if one or more of the selected items demonstrates a lack of homogeneity.

9. Calculations

9.1. See Appendix.

10. Uncertainty of Measurement

10.1. Not applicable (this SOP covers this aspect).

11. Limitations

11.1. Not applicable

12. Documentation

12.1. FCU Examination Worksheets
12.2. FCU Report of Results
12.3. Measurement Uncertainty Estimation Form

13. References

13.1. Forensic Science Laboratory (FSL) Quality Assurance Manual (Current Version)
13.2. DFS Departmental Operations Manuals (Current Versions)
13.3. FSL Laboratory Operations Manuals (Current Versions)

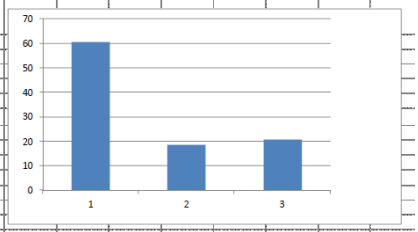
14. Appendix - Example Uncertainty Calculations

14.1. Example calculation for determining uncertainty for weight measurement

Measurement Uncertainty Estimation Form

Measurement:	Calibration of Force-Measuring Instruments
Range of measurement values:	0.00 g to 320.00 g
Procedure name and revision:	Uncertainty of Measurement for Benchtop Analytical Balances
Estimation prepared by:	ASCLD/LAB Guidance Document Annex B
Date Prepared:	12/6/2018

Line Item	Uncertainty Component	Value	Units	Distribution	Type	Divisor	Degrees Freedom (n-1)	Standard Uncertainty	Component Contribution %
1	Measurement Process Reproducibility	0.00019	g	A	normal	1	55	0.00019	61
2	Resolution of Measurement	0.00010	g	B	rectangular	1.73	∞	0.00006	19
3	Balance Calibration Uncertainty	0.00013	g	B	normal	2.00	∞	0.00007	21
	Combined Standard Unc	u_c						0.0002	100
df>100	Expanded Unc in grams	U (k=2)						0.0004	
	Expanded Unc in grams	U (k=3)						0.0006	
	Reported Uncertainty:	**0.0004**		k=2	95.45% confidence				
	Reported Uncertainty:	0.0006		k=3	99.7% confidence				



NOTE: Regardless of the number of digits that are showing in a cell, Excel carries the maximum number of significant figures in the background and will use the entire number for further calculations

Notes/Assumptions:

1. Measurement Process Reproducibility - this entry is a the highest obtained estimation for uncertainty components listed in Step 3 as Type A method of evaluation assumes a normal distribution.
2. Balance Calibration Uncertainty obtained from the Mettler Toledo calculated uncertainty value.
3. All analysts were involved in the measurement of uncertainty determination
4. The major components of uncertainty of measurement come from the balance itself and human error
   Sample Weights: 2g, 0.1g, 0.01g

Revision: 2018

14.2. Example calculation for determining uncertainty for heroin purity

Measurement Uncertainty Estimation Form

Measurement:	Purity Determination by GC-FID
Range of measurement values:	Percentage (0-100%)
Procedure name and revision:	Uncertainty of Measurement for Quantitative Purity Determinations
Estimation prepared by:	Brandon Jones
Date Prepared:	3/12/2019

Line Item	Uncertainty Component	Value	Units	Distribution	Type	Divisor	Degrees Freedom (n-1)	Standard Uncertainty	Component Contribution %
1	Process Reproducibility	1.400%	%	A	normal	1		0.0140	50.9
2	Balance Uncertainty	0.600%	%	A	normal	3.00	∞	0.0020	7.3
3	Volumetric Uncertainty	0.300%	%	A	normal	2.00	∞	0.0015	5.5
4	Standard Purity Uncertainty	2.000%	%	A	normal	2.00	∞	0.0100	36.4
	Combined Standard Unc	u_c						0.0174	64
df>100	Expanded Unc in % of purity	U (k=2)						0.0348	
	Expanded Unc in % of purity	U (k=3)						0.0522	
	Reported Uncertainty:	**3.477%**		k=2	95.45% confidence				
	Reported Uncertainty:	5.216%		k=3	99.7% confidence				

NOTE: Regardless of the number of digits that are showing in a cell, Excel carries the maximum number of significant figures in the background and will use the entire number for further calculations

Notes/Assumptions:

1. Measurement Process Reproducibility - this entry is a the highest obtained estimation for uncertainty components listed in Step 3 as Type A
2. Balance Uncertainty obtained from the calculated uncertainty of measurement value.
3. Volumetric uncertainty is calculated from the uncertainty associated with a 5mL volumetric flask.
4. The major components of uncertainty of measurement come from the instrument itself and human error.

Revision: Initial